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Enhanced corrosion protection of AZ31 magnesium alloy by duplex plasma electrolytic oxidation and polymer coatings

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Key words

Magnesium alloy; Plasma electrolytic oxidation; Polymer coating; Corrosion resistance; Electrochemical impedance spectroscopy.

Abstract

The corrosion behaviour of an AZ31 magnesium alloy coated with plasma electrolytic oxidation (PEO), polymer and a combination of was assessed in by electrochemical impedance spectroscopy (EIS) in both 0.1 M NaCl solution. The polymer coating was applied on a just cleaned surface (without any special preparation) and also on PEO treated magnesium substrate. While the polymer coated and the mere PEO treated magnesium alloy specimens lasted less than 50 hours in the EIS tests, the specimens with the duplex coating (PEO + polymer) successfully resisted 1000 hours in the EIS tests without showing any degradation. The improved adhesion of the polymer coating in presence of the PEO layer and the effective sealing of the pores in the PEO coating with the polymer were responsible for the enhanced corrosion resistance. The synergistic beneficial effect of the duplex coatings on the corrosion behaviour was also witnessed in the salt spray tests.

Introduction

Due to their poor corrosion resistance magnesium alloys require a surface treatment or modification for weathering corrosive environments [1-2]. A variety of surface treatments are contemplated for protection of magnesium alloys, which include conversion coatings, plasma anodization, physical vapour deposition and electro/electroless plating [3-6]. The employment of plasma electrolytic oxidation (PEO) treatment for improving the corrosion resistance and tribological behaviour of magnesium alloys is gaining importance day by day [7-8]. Some recent research attempts addressed the application of polymer coatings for magnesium alloys [9-10], but in both the papers the evaluation was restricted to short test durations. The role of conducting polymers as coatings for magnesium alloys was also documented in literature [11-13]. Beneficial effects of duplex coatings in the form of PEO-physical vapour deposition, PEO-electroless nickel and PEO-composite coatings on the wear and corrosion behaviour of magnesium alloys have been reported recently [14-16].

Even though the corrosion resistance offered by the PEO coatings, as assessed by short term electrochemical tests, were reported to be good, in the long term exposure studies the PEO coatings were found to fail [17]. The degradation was believed to be on account of the seepage of electrolyte through the pores in the PEO coating to the

1 surface of the magnesium substrate, thus causing the corrosion damage. One of the
2 possible ways of improving the long term corrosion performance would be to have a
3 polymer coating over the PEO layer. To the best of the knowledge of the authors, there
4 is no published information on the duplex coatings consisting of PEO coating and
5 polymer layer. As it could be industrially relevant for a wide variety of applications
6 ranging from automotive to household appliances, an attempt has been made in this
7 investigation to understand the effect of a duplex coating (PEO + polymer) on the long
8 term corrosion performance of AZ31 magnesium alloy substrates in chloride
9 environments.
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12 **Experimental**

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14 Magnesium alloy sheet corresponding to AZ31 with a nominal chemical composition
15 of 3 wt% Al, 1 wt% Zn and balance Mg was used in this investigation. Specimens of
16 size 50 mm x 20 mm x 3 mm were used for all the experiments. They were prepared by
17 polishing up to 2500 grit emery, cleaned with acetone and then plasma electrolytic
18 oxidation treated in an electrolyte consisting of 5 g potassium hydroxide and 10 g of
19 sodium silicate in 1 liter of distilled water. The PEO treatment was carried out at
20 a current density of 15 mA·cm⁻² up to a final voltage of 440 V. The treatment was
21 continued until the applied current reached a value 0.05 A at this constant final voltage.
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25 For polymer coating on the PEO coated magnesium alloy specimens, the poly(ether
26 imide) Ultem 1000[®] with a chemical structure shown in **Figure 1** was employed.
27 Polymer solutions were prepared in dichloromethane (CH₂Cl₂) of analytical grade with a
28 concentration of 1 wt.-% to control the coating thickness and to also ensure the
29 penetration into the pores of the PEO coating by the viscosity of the solution.
30 Specimens were prepared by a chemical cleaning in an aqueous solution consisting of
31 20% acetic acid and 8% of calcium nitrate followed by a thorough rinse in distilled water.
32 These specimens were then dried with warm air to remove the traces of moisture before
33 the application of polymer coating, which was applied by dipping the specimens into the
34 polymer solution at ambient temperature, followed by drying in a vacuum oven (10
35 mbar) at 40°C for 4 hours. For reference, magnesium alloys in the just-cleaned
36 condition were also given the same type of polymer coating under similar processing
37 conditions.
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42 The cross-sections of the PEO, polymer and PEO + polymer coated specimens were
43 prepared by conventional metallographic technique by polishing successively in 500,
44 1200 and 2500 grit emery papers, followed by final disc polishing using colloidal silica
45 suspension (OP-S). Optical micrographs were taken in the as-polished condition
46 (without etching) to reveal the thickness and cross-sectional features. The surface
47 morphology of the PEO coated specimen was examined in a Cambridge Stereoscan
48 scanning electron microscope.
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51 The corrosion potential of the specimens was measured for a period of 1800 s before
52 performing the electrochemical tests. Electrochemical impedance spectroscopy (EIS)
53 studies were made in an ACM Gill AC potentiostat/galvanostat FRA, using a three
54 electrode cell, with an applied amplitude of 10 mV in the frequency range 0.01 Hz to
55 30,000 Hz at the free corrosion potential. The uncoated specimens were prepared by
56 polishing up to 2500 grit emery, while the PEO coated and polymer coated specimens
57 were used in the as-coated condition. Experiments were performed at ambient
58 temperature (21±2°C), in non-deaerated 0.1 M NaCl solution in the as-prepared
59 condition. Free corrosion potential measurements were made for 120 s before each
60 impedance measurement. Long term impedance measurements were made at regular
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1 and defined intervals on these PEO coated and polymer coated specimens until the
2 failure of the coating was observed. For the case of the duplex coatings (PEO +
3 polymer) the experiments were continued up to 1000 hours. The untreated, PEO
4 coated and PEO + polymer coated specimens were also subjected to salt spray tests in
5 accordance with ASTM B117 standard.
6

8 Results and Discussion

10 PEO coating

11 An optical micrograph showing the cross-section of the PEO coated specimen is
12 presented in **Figure 2(a)**, the thickness of the PEO coating was estimated to be
13 $12 \pm 2 \mu\text{m}$. The wavy interface between the coating and the matrix characteristic of PEO
14 integral coatings is evident in this micrograph. **Figure 2(b)** shows the surface
15 morphology of the PEO coated specimen revealing the presence of pores, with pore
16 diameters in the range of $0.5 \mu\text{m}$ to $10 \mu\text{m}$. The spark sizes during plasma processing in
17 the initial stages up to around 400 V were very fine and they lived a rather short life.
18 However, with a further increase in the voltage up to 440 V, the sparks were found to
19 grow to larger sizes, with relatively long life-time compared to those at the lower
20 voltages. The different sizes of pores developed on the surface are attributed to the
21 varied sizes of sparks during processing. Further, due to the high voltage discharge
22 conditions, crack-like lenticular shaped pores were observed on this surface. The
23 surface roughness measurements made using a Hommel tester showed a mean
24 roughness value (R_a) of $0.84 \pm 0.08 \mu\text{m}$ for the PEO coated specimen. The roughness of
25 the untreated AZ31 magnesium alloy substrate was $0.12 \pm 0.02 \mu\text{m}$ before the PEO
26 treatment. It is not surprising that the PEO treatment resulted in a rougher coating which
27 is attributed to the coarse sparks that had resulted in the formation of large chunks of
28 ceramic oxide deposits and the resultant thick coating with large sized pores.
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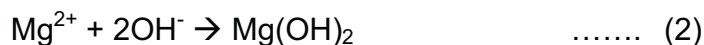
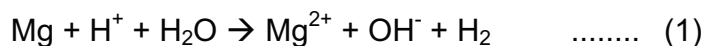
33
34 The EIS spectra of the PEO coated specimen in 0.1 M NaCl solution is presented in
35 **Figure 3a**. The PEO coating has exhibited an overall impedance of $9.1 \times 10^5 \Omega \cdot \text{cm}^2$
36 after 30 minutes of exposure to the electrolyte. The resistance was found to be slightly
37 more in the measurements made after 2 h and 5 h, registering values of $1.1 \times 10^6 \Omega \cdot \text{cm}^2$
38 and $1.4 \times 10^6 \Omega \cdot \text{cm}^2$. However, with further exposure to the electrolyte, the resistance
39 dropped to $7.9 \times 10^5 \Omega \cdot \text{cm}^2$ and $3.1 \times 10^5 \Omega \cdot \text{cm}^2$ after 25 and 50 hours of exposures,
40 respectively.
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44 The potential measurements made before the EIS experiments also corroborate the
45 above results (**Figure 3b**). The free corrosion potential of the PEO coated specimen
46 was observed to shift towards the noble direction after 2, 5 and 10 hours of exposures
47 compared to the values observed in the specimen exposed for 30 minutes. On the
48 other hand, the potential drifted towards the active direction in the 25 hours test,
49 indicating the start of degradation of the coating. This is reflected in the impedance plot,
50 with a drop in resistance from $1.1 \times 10^6 \Omega \cdot \text{cm}^2$ registered in the test after 10 hours to
51 $7.9 \times 10^5 \Omega \cdot \text{cm}^2$ after 25 hours of exposure. It is evident that the dissolution of
52 magnesium owing to the seepage through pores had caused the formation of corrosion
53 products ($\text{Mg}(\text{OH})_2$) initially, thus showing the effect of passivation [17-18].
54 Subsequently, due to the continued corrosion process, the coating-substrate interface
55 had deteriorated, resulting in the shift of potential towards the active side, which is
56 reflected in the impedance behaviour as well. The optical macrograph of the EIS tested
57 surface of the PEO coated specimen shown in **Figure 4** reveals the extent of corrosion
58 attack. It is obvious that the localised corrosion that had occurred on the PEO surface
59 had damaged the coating at a few places.
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Polymer coating

The optical micrograph showing the cross-section of the AZ31 magnesium alloy with the polymer coating is depicted in **Figure 5**. The coating was found to be uniform on the surface with a thickness of $25 \pm 2 \mu\text{m}$. **Figure 6(a)** shows the EIS spectra of the polymer coated magnesium alloy substrate obtained after different exposure times, and the corresponding free corrosion potential measurements are shown in **Figure 6(b)**. It is well known that the free corrosion potential is governed by the nature/type of coating, thickness and the extent of defect level. The better and compact the coating, the nobler would be the corrosion potential. Sathiyarayanan et al. [19] have reported free corrosion potentials nobler than -0.8 V vs. SCE for polyaniline-acrylic coated ZM21 magnesium alloy substrates. However, the coating thickness was nearly $100 \mu\text{m}$ in that investigation. The free corrosion potential of the polymer coated specimen in the current investigation was around $-1280 \text{ mV vs. Ag/AgCl}$ after 30 minutes of exposure and was found to drift towards the positive side with increase in exposure time up to 10 hours, registering a value of around $-1140 \text{ mV vs. Ag/AgCl}$. The trend in the drifting of potential towards the nobler side was similar to that observed in the case of PEO coated specimen; however, the potentials were slightly in the nobler side for the polymer coated specimen.

In the presence of polymer coating, an impedance value of $2.9 \times 10^7 \Omega\text{-cm}^2$ was observed in the EIS test after 30 minutes. Afterwards, the resistance was found to increase to $3.6 \times 10^7 \Omega\text{-cm}^2$ and $5.3 \times 10^7 \Omega\text{-cm}^2$ in the tests after 2 hours and 10 hours, respectively. The resistance was found to drop to a value of $2.8 \times 10^6 \Omega\text{-cm}^2$ after 25 hours and to a still lower value of $9.0 \times 10^3 \Omega\text{-cm}^2$ after 50 hours, which is already close to the bare AZ31 magnesium alloy substrate in this solution. Even though the polymer coating appeared to be dense and compact, it appears that the diffusion/permeation of electrolyte ions through the polymer film to reach the substrate and the consequent dissolution of the Mg substrate surface had plausibly led to the passivation. It is well known that the electrochemical reactions occur at the surface of magnesium substrate due to prolonged exposure and result in the formation of hydrogen and magnesium hydroxide:



The formation of a passive film at the interface was responsible for the increase in resistance values after 2 and 10 hours of exposure. Upon further exposure, the generation of more amounts of hydrogen and formation of $\text{Mg}(\text{OH})_2$ had led to the deterioration of the coating. The combined effect of the increase in hydrogen pressure with time underneath the polymer film and the formation of magnesium hydroxide on the surface of the substrate seem to have resulted in blistering of polymer film after 50 hours of EIS testing. The corrosion damage underneath the polymer film (white corrosion products) and the cracking of polymer film as a result of hydrogen and also the piled up stress due to $\text{Mg}(\text{OH})_2$ formation are evident in **Figure 7**. The infrared spectroscopy measurements in an associated work [10] showed bonds of OH and $\text{Mg}(\text{OH})_2$ at wave numbers above 3500 cm^{-1} , confirming the corrosion attack at the magnesium substrate. Kannan et al. [9] have reported a very similar degradation behaviour of poly (4,4'-diphenyl-ether-1,3,4-oxadiazole) coated AM50 magnesium alloy substrate.

Duplex coating (PEO + polymer)

An attempt was made to examine the mere polymer and PEO + polymer coated specimens at a higher magnification in the scanning electron microscope to assess the bonding at the substrate/polymer interface and PEO/polymer interface in the respective cases and also the filling of pores in the PEO coating by the polymer. However, the polymer film was found to be destroyed even at an accelerating voltage of 3 kV and hence this assessment could not be accomplished. The optical micrograph showing the cross-section of the PEO + polymer coated magnesium alloy substrate is presented in **Figure 8**. The polymer coating thickness of around 25 μm in the duplex coating was the same as that observed in the mere polymer coated specimen.

The EIS data obtained for the PEO + polymer coated specimen were fitted using a simple electrochemical circuit as depicted in **Figure 9** and the derived electrochemical data are reported in **Table 1**. The free corrosion potentials and the electrochemical impedance behaviour of the PEO + polymer coated specimen are presented in **Figures 10(a)** and **(b)**, respectively. This specimen had registered a free corrosion potential of around -1210 mV vs. Ag/AgCl after 30 minutes of exposure to the electrolyte. Upon further exposure, the passivation effect was observed for this specimen too, and a potential value close to -920 mV vs. Ag/AgCl was registered after 50 hours. The potential values were around -1000 mV vs. Ag/AgCl in the tests after 100, 250 and 500 hours and dropped to around -1100 mV vs. Ag/AgCl after 1000 hours exposure. However, this value was still nobler compared to the value registered in the test after 30 minutes of exposure. It should be pointed out here that the potentials of the mere polymer coated and the PEO coated specimens were found to drop to around -1400 mV vs. Ag/AgCl after 50 hours of exposure/EIS tests. A much nobler potential even after 1000 hours for the PEO + polymer coated specimen under similar testing conditions thus demonstrates the stability of this duplex coating in this electrolyte.

Table 1 Electrochemical data obtained using the equivalent circuit for the PEO + polymer coated specimen

Exposure time, hours	Capacitance of polymer coating $C_P, \text{F}\cdot\text{cm}^{-2}$	Resistance of polymer coating $R_P, \Omega\cdot\text{cm}^2$	Capacitance of PEO coating, $C_{\text{PEO}}, \text{F}\cdot\text{cm}^{-2}$	Resistance of PEO coating $R_{\text{PEO}}, \Omega\cdot\text{cm}^2$
0.5	5.0×10^{-8}	1.6×10^6	4.6×10^{-10}	4.9×10^6
50	2.0×10^{-8}	6.7×10^6	4.9×10^{-10}	1.2×10^7
100	2.9×10^{-8}	3.6×10^6	5.0×10^{-10}	1.2×10^7
250	1.2×10^{-8}	5.4×10^6	5.6×10^{-10}	9.4×10^6
500	8.3×10^{-9}	7.2×10^6	5.4×10^{-10}	1.0×10^7
1000	3.8×10^{-9}	6.6×10^6	6.0×10^{-10}	4.6×10^6

From Figure 10(b) and Table 1 it is evident that the resistances offered by the PEO and polymer layers were nearly the same, resulting in an overall resistance value of around $10^7 \Omega\cdot\text{cm}^2$ in the entire duration (1000 hours) of the EIS tests. In the case of mere polymer coating, and mere PEO coating, too, the resistances offered by the respective layers were of the same magnitude in the initial stages, say up to 10 hours. However, with long term exposure to the electrolyte, the aforementioned mono-coatings deteriorated, resulting in complete failure in about 50 hours. It was found that the mere polymer coating failed due to the seepage of electrolyte by diffusion of electrolyte ions in the film, and resultant corrosion process/hydrogen evolution/ $\text{Mg}(\text{OH})_2$ formation at the

1 coating-substrate interface. In the duplex coating, the polymer film had apparently filled
2 the pores in the PEO coating, and thus effectively sealed the PEO coating. Thus, the
3 PEO and polymer coatings, in combination, have synergistically provided a superior
4 corrosion protection to the magnesium alloy substrate in the long term EIS tests. It is
5 believed that this is seemingly due to the improved adhesion of the polymer by the
6 rough PEO surface. The optical macrograph of the PEO + polymer coated specimen
7 after 1000 hours of EIS tests shown in **Figures 11** shows no signs of visible damage on
8 the surface, and this further supplements the electrochemical data.
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12 The untreated, PEO coated, polymer coated and PEO + polymer coated specimens
13 were subjected to salt spray tests as per ASTM B117. The optical macrographs of the
14 specimens with and without the coatings after the tests are shown in **Figures 12 (a)-(d)**.
15 The untreated and polymer coated specimens were found to undergo corrosion damage
16 within 24 hours of exposure, and the extent of damage on the respective surfaces after
17 48 hours of exposure can be seen in Figures 12(a) and (b). The electrolyte had seeped
18 through the polymer coating causing it to flake-off with the development of corrosion
19 products underneath the polymer film. This clearly suggests that a combination of
20 solvent/alkali/acid cleaning alone is not sufficient as a surface preparation for the
21 coating of polymer on magnesium substrates. The mere PEO coating was found to
22 pass 48 hours of salt spray test without much of surface degradation. However, the
23 corrosion damage was found to develop on both these specimens in the form of
24 localised attack viz., pits (marked by arrows in Figures 12(c)). On the other hand, the
25 PEO + polymer coated specimens passed 300 hours of test duration without showing
26 any signs of degradation (Figure 12(d)).
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32 **Conclusions**

33
34 The application of PEO and polymer coatings offers good corrosion resistance to the
35 AZ31 magnesium substrate as evidenced in the short-term EIS tests. However, long
36 term exposures to the corrosive environment lead to degradation of these coatings
37 resulting in failure within 50 hours. The seepage of electrolyte through the defects in
38 these coatings was responsible for the corrosion at the coating-substrate leading to
39 damage. The duplex coatings consisting of PEO and polymer layers offered a much
40 superior corrosion resistance in the long term EIS tests and salt spray tests without
41 showing any signs of any visible corrosion damage on the tested surfaces. This is
42 attributed to the effective sealing of pores in PEO coating by the polymer, thus efficiently
43 protecting the substrate from coming in contact with the corrosive test environment.
44 The PEO coating in the duplex coating not only acts as a second barrier, but also
45 provides and improved adhesion to the polymer layer, thus preventing the de-lamination
46 failure of the polymer. Furthermore, the polymer layer reduces the water/chloride ions at
47 the weaker contact area (magnesium substrate/PEO coating interface), thus giving the
48 whole system more stability.
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54 **Acknowledgement**

55
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Figure Captions

- 1
2
3 Figure 1 Chemical structure of a repetition unit of poly(ether imide) Ultem 1000®
4
5 Figure 2 Microstructural features of the PEO coated AZ31 magnesium alloy
6 (a) Optical micrograph showing the cross-section
7 (b) Scanning electron micrograph showing the surface morphology
8
9
10 Figure 3(a) EIS spectra of the PEO coated AZ31 magnesium alloy in 0.1 M NaCl solution
11 after different durations of exposure
12
13 Figure 3(b) Potential measurements of the PEO coated AZ31 magnesium alloy in 0.1M NaCl
14 solution after different times of exposure
15
16 Figure 4 Optical macrograph of the PEO coated specimen surface showing the extent of
17 corrosion damage after 50 hours of impedance measurements in 0.1 M NaCl
18 solution
19
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21 Figure 5 Optical micrograph showing the cross-section of polymer coated AZ31
22 magnesium alloy
23
24 Figure 6(a) EIS spectra of the polymer coated AZ31 magnesium alloy in 0.1 M NaCl solution
25 after different durations of exposure
26
27 Figure 6(b) Potential measurements of the polymer coated AZ31 magnesium alloy in 0.1 M
28 NaCl solution after different times of exposure
29
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31 Figure 7 Optical macrograph of the polymer coated specimen surface showing the
32 corrosion damage and blisters in the polymer coating after 50 hours of
33 impedance measurements in 0.1 M NaCl solution
34
35 Figure 8 Optical micrographs showing the cross-sections of PEO + polymer coated AZ31
36 alloy
37
38
39 Figure 9 Electrochemical circuit employed for fitting the impedance data
40 R_s → Resistance of solution
41 C_p, C_{PEO} → Capacitance of polymer coating and PEO coating
42 R_p, R_{PEO} → Resistance of polymer coating and PEO coating
43
44 Figure 10(a) Potential measurements of the magnesium alloy with the duplex coating (PEO +
45 polymer) in 0.1 M NaCl solution after different durations of exposure
46
47 Figure 10(b) EIS spectra of the magnesium alloy with the duplex coating (PEO + polymer) in
48 0.1 M NaCl solution after different durations of exposure
49
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51 Figure 11 Optical macrograph of PEO + polymer coated specimen surface after 1000 hours
52 of impedance measurements in 0.1 M NaCl solution
53
54 Figure 12 Macrographs of the AZ31 magnesium alloy specimens with and without coatings
55 after salt spray tests
56 (a) Untreated, (b) polymer, (c) PEO coated specimens → after 48 hours of
57 exposure
58 (d) PEO + polymer coated specimen → after 300 hours of exposure
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Figure 1
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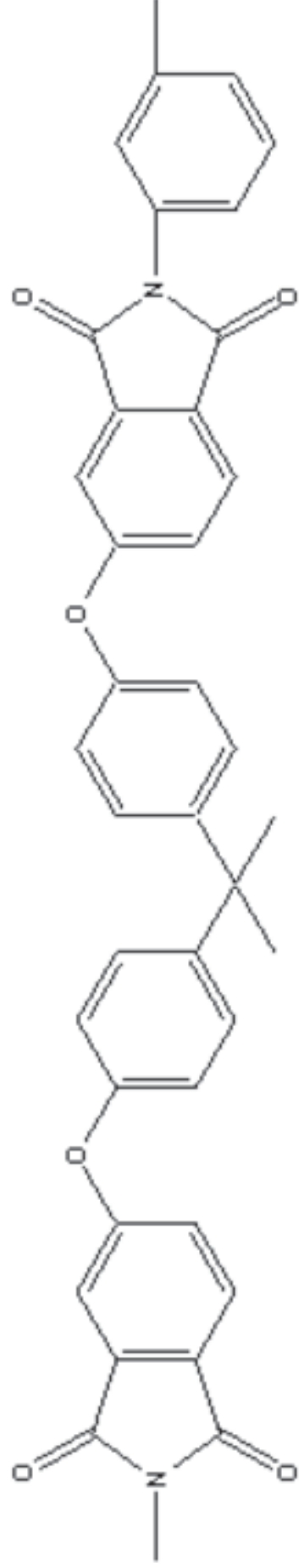


Figure 2a
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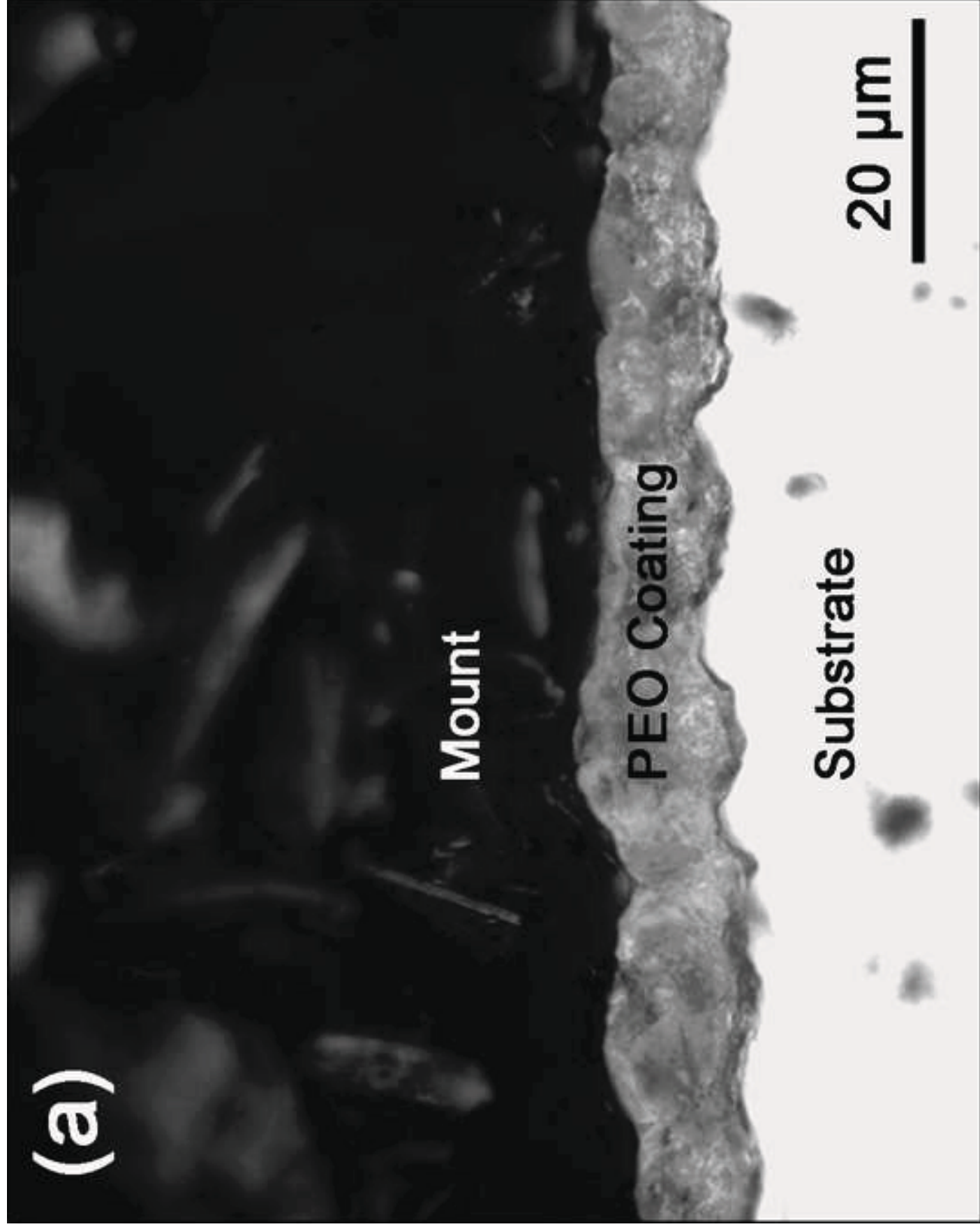


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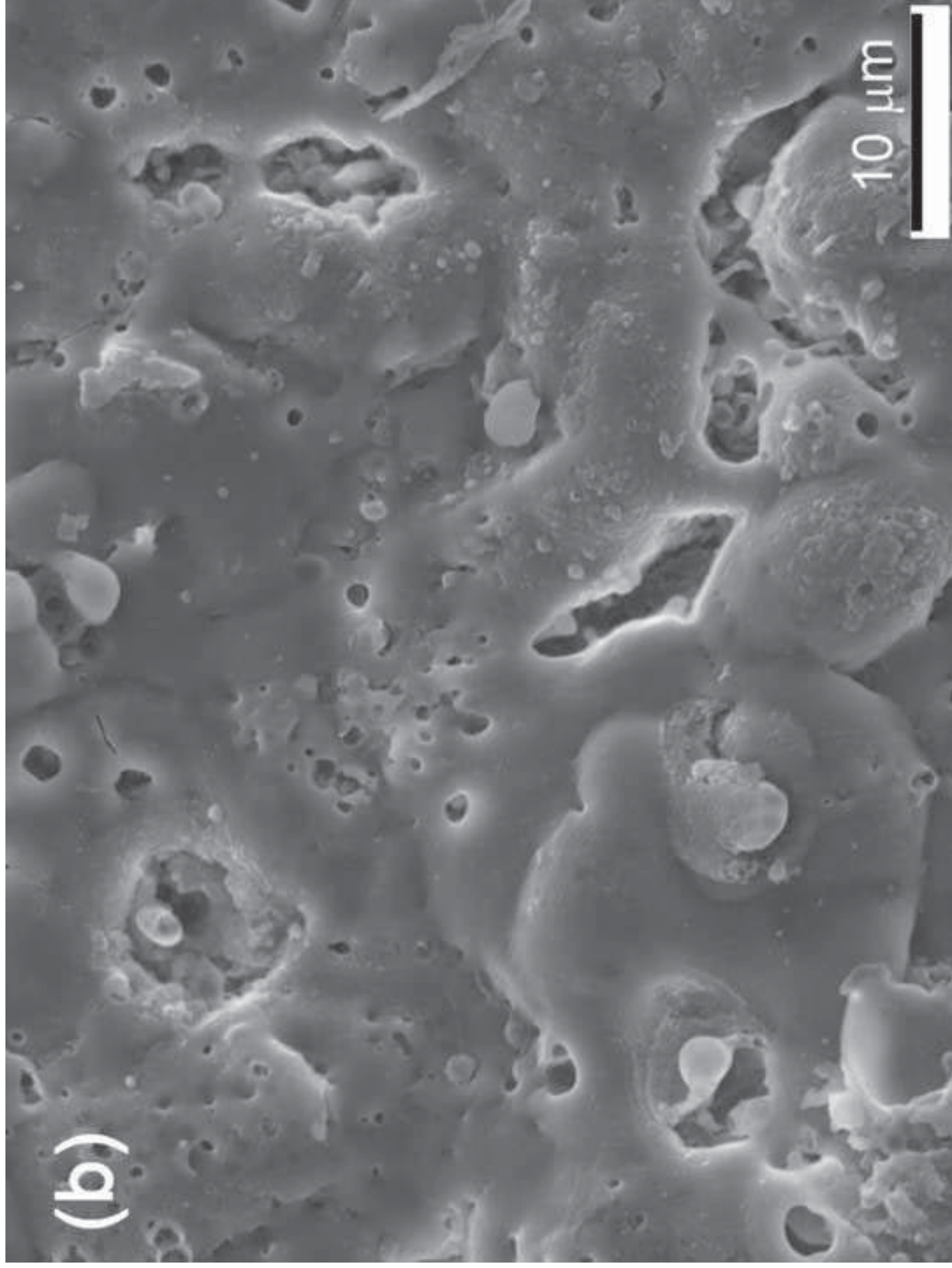


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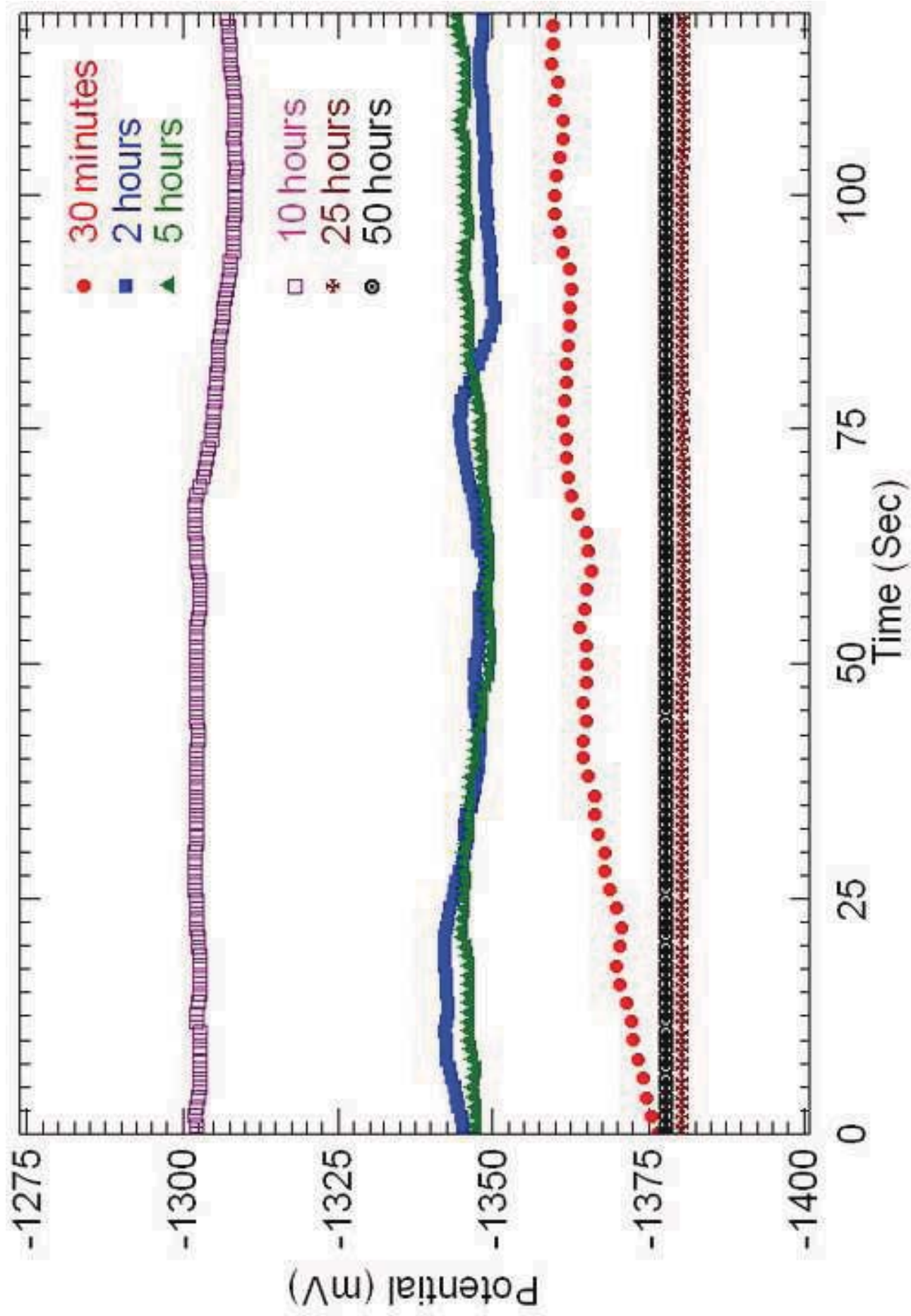


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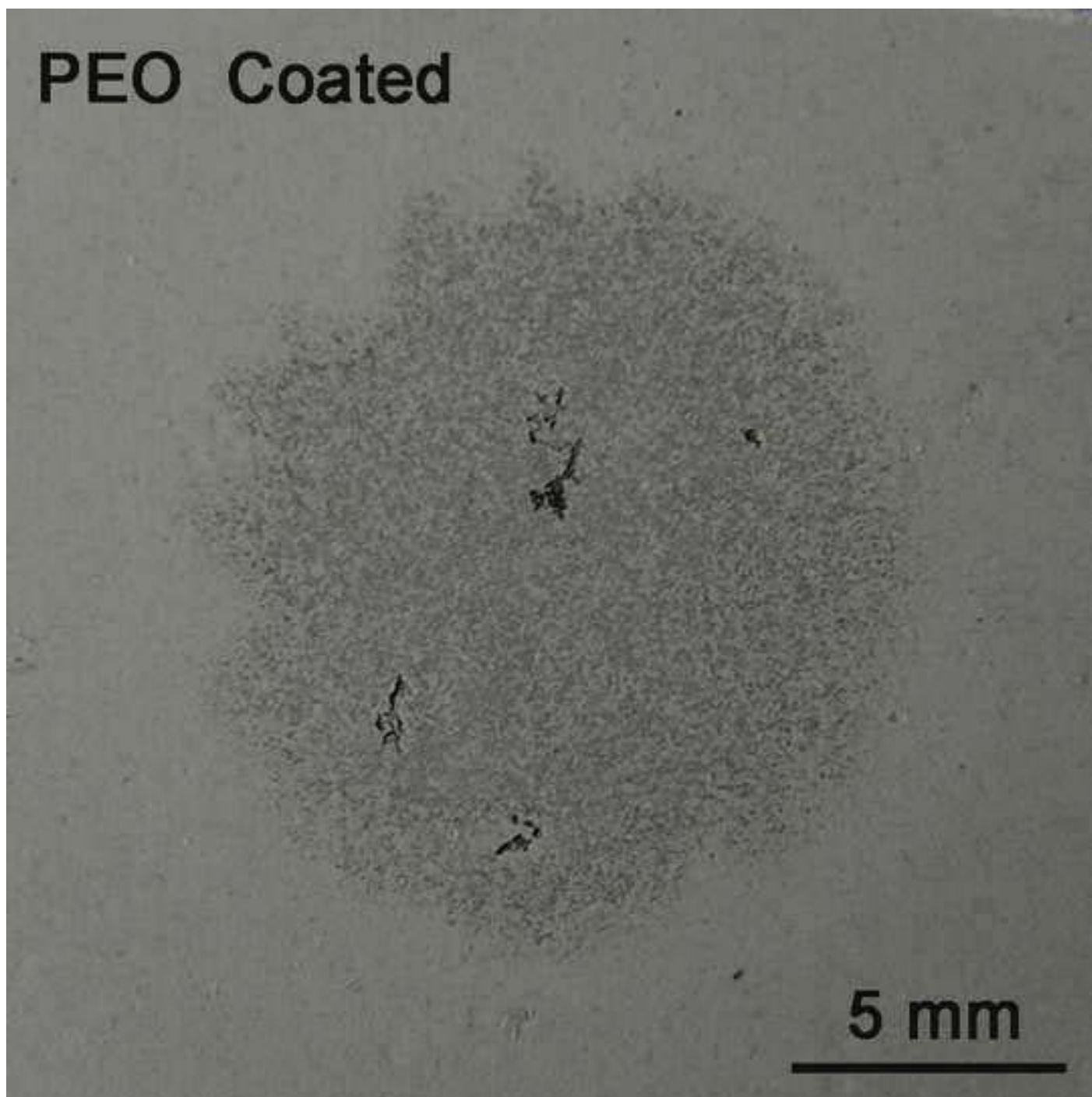


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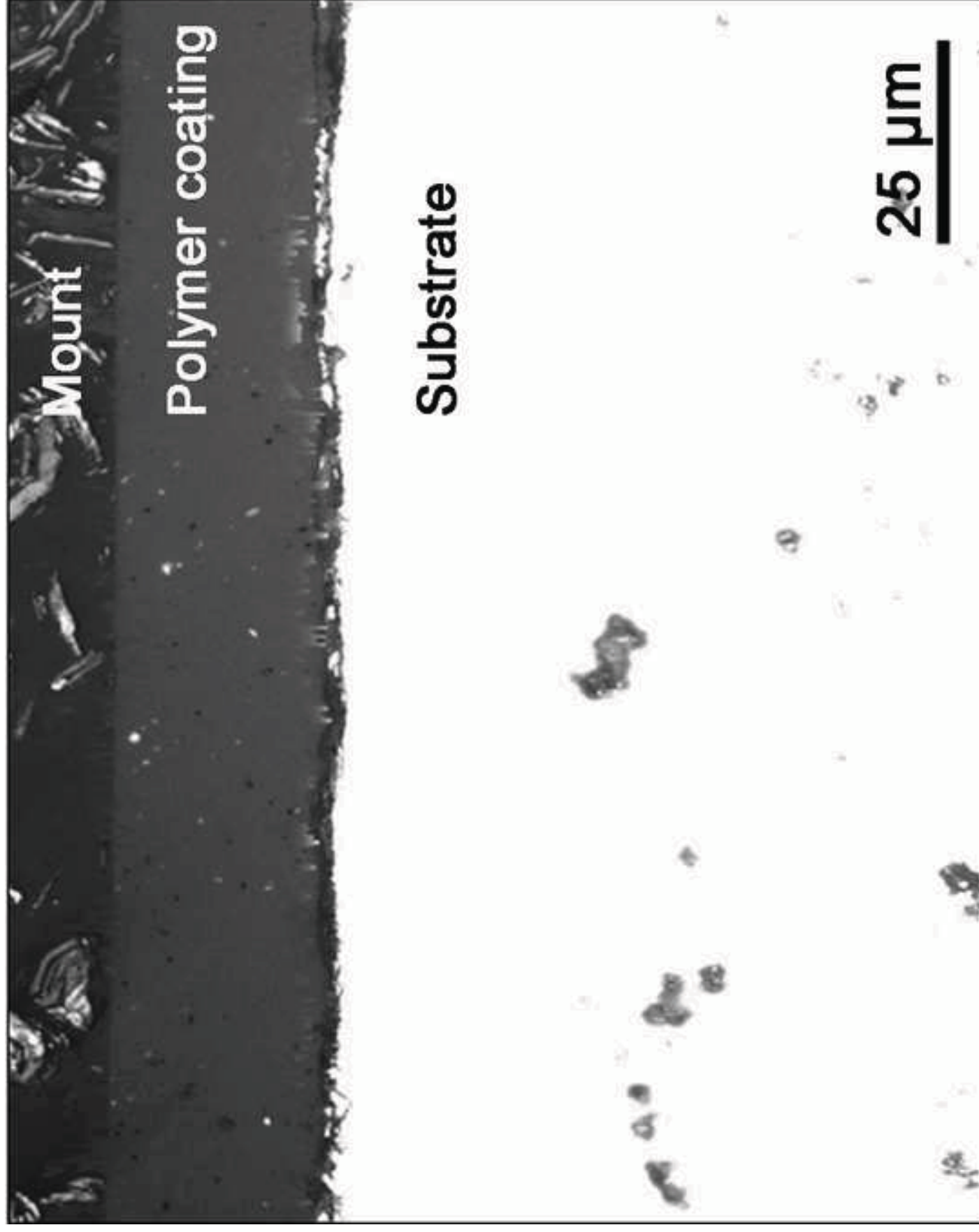


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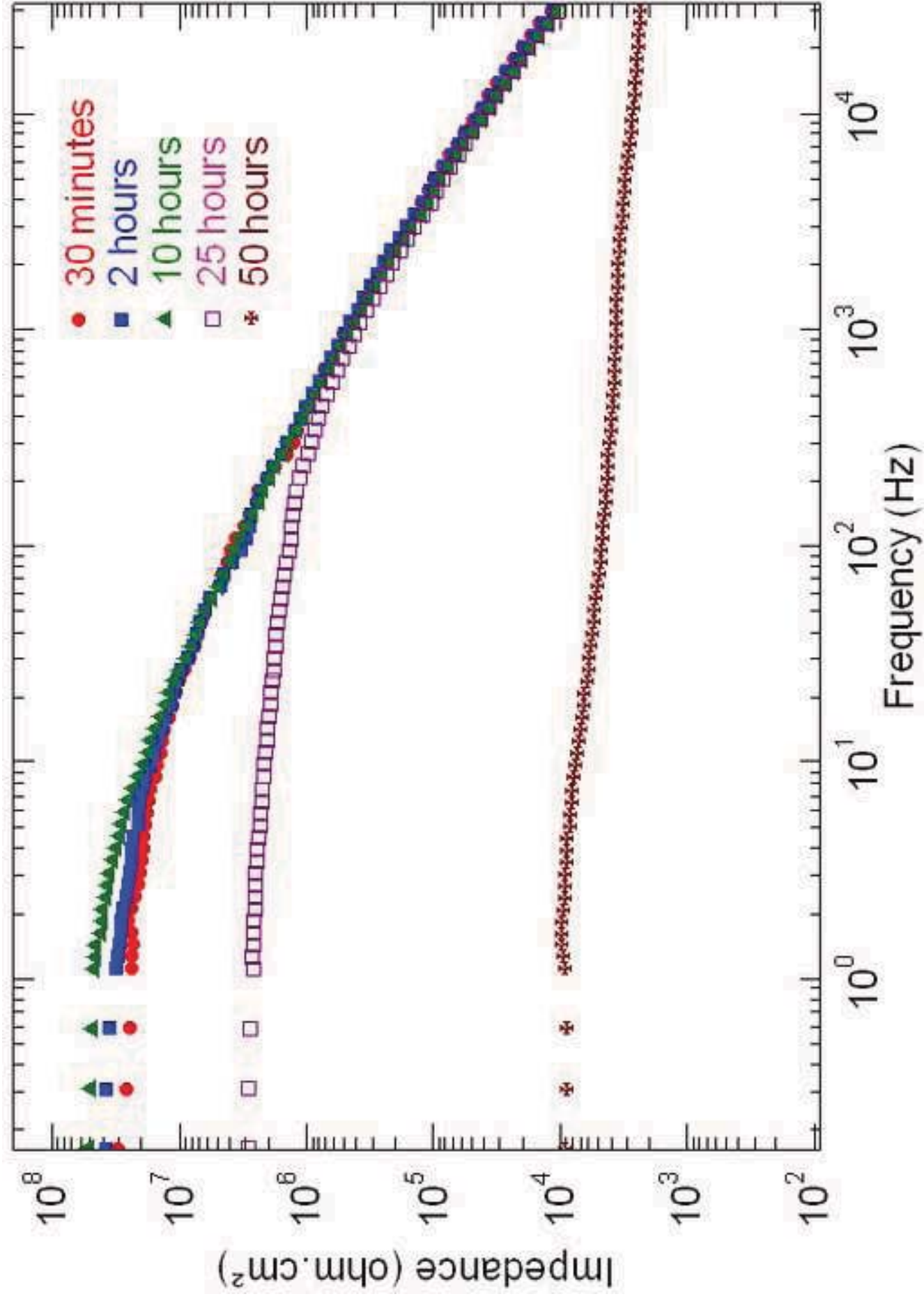


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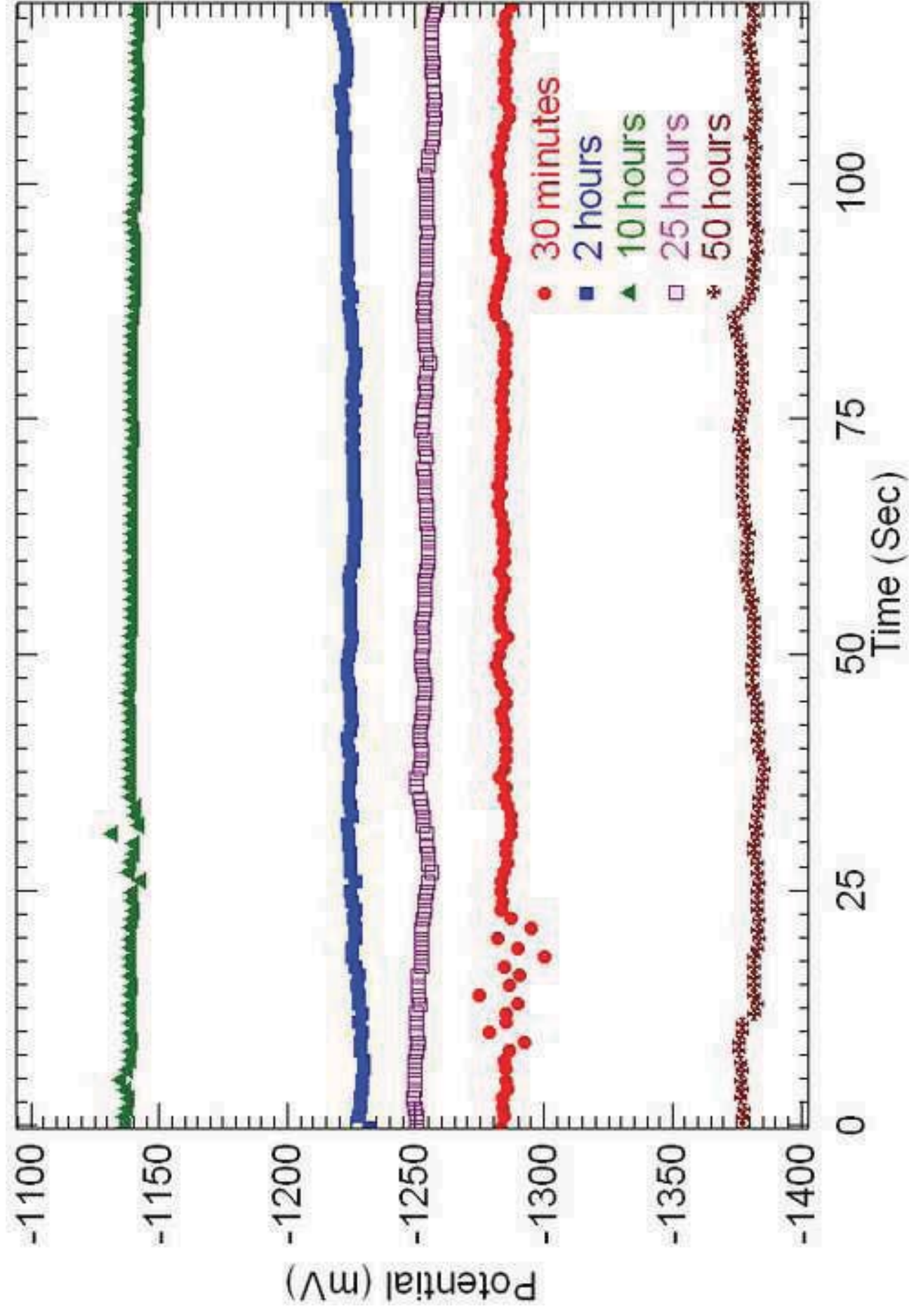


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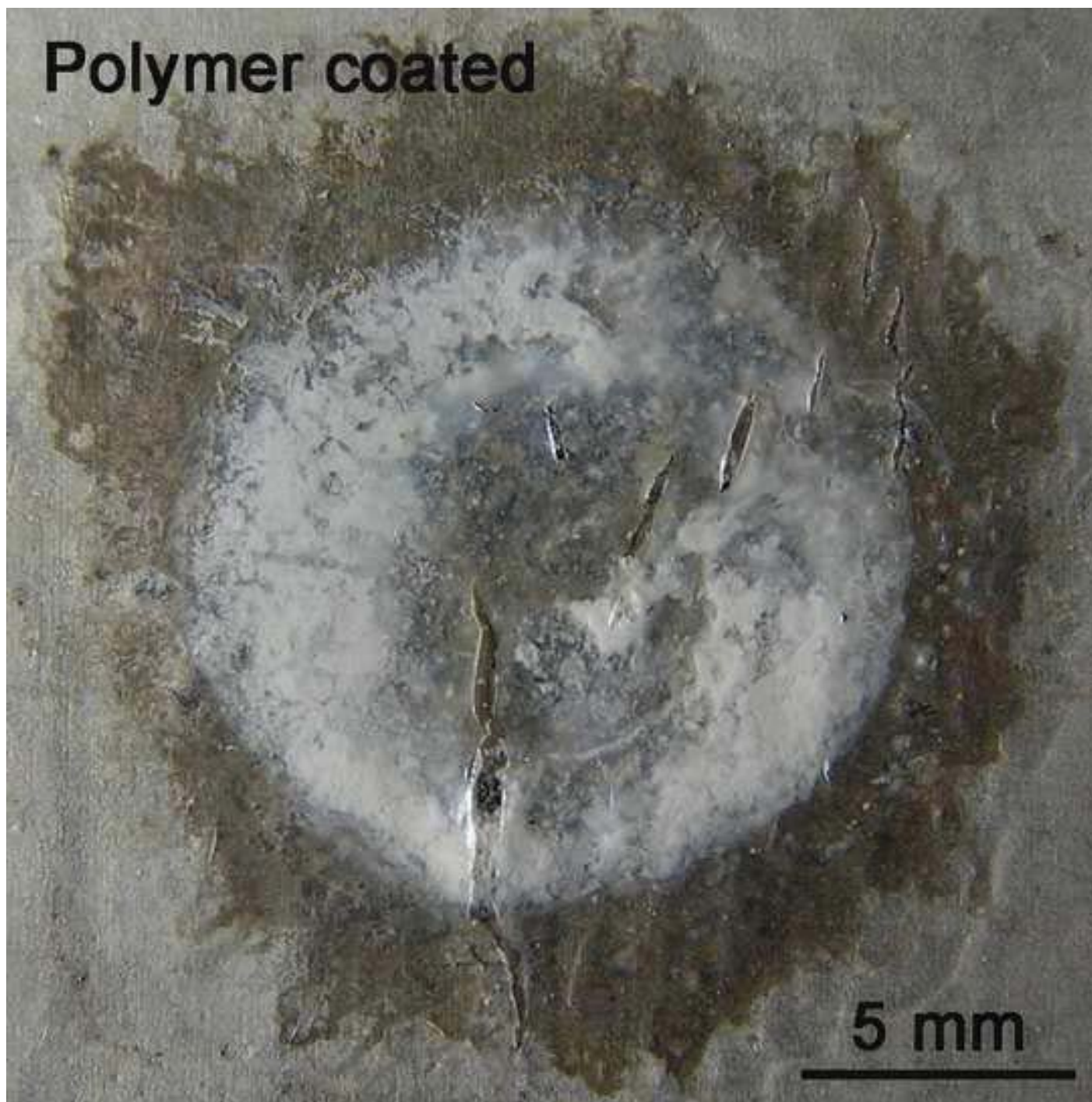


Figure 8
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Figure 9
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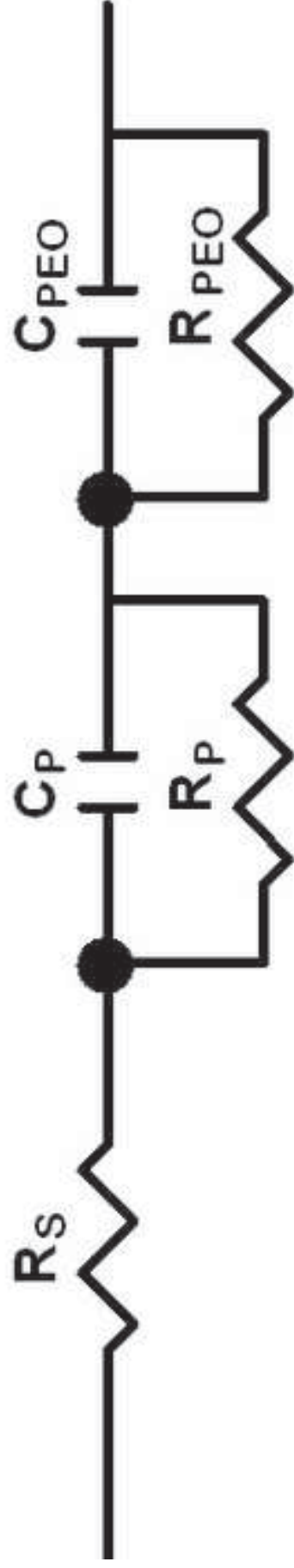


Figure 10a

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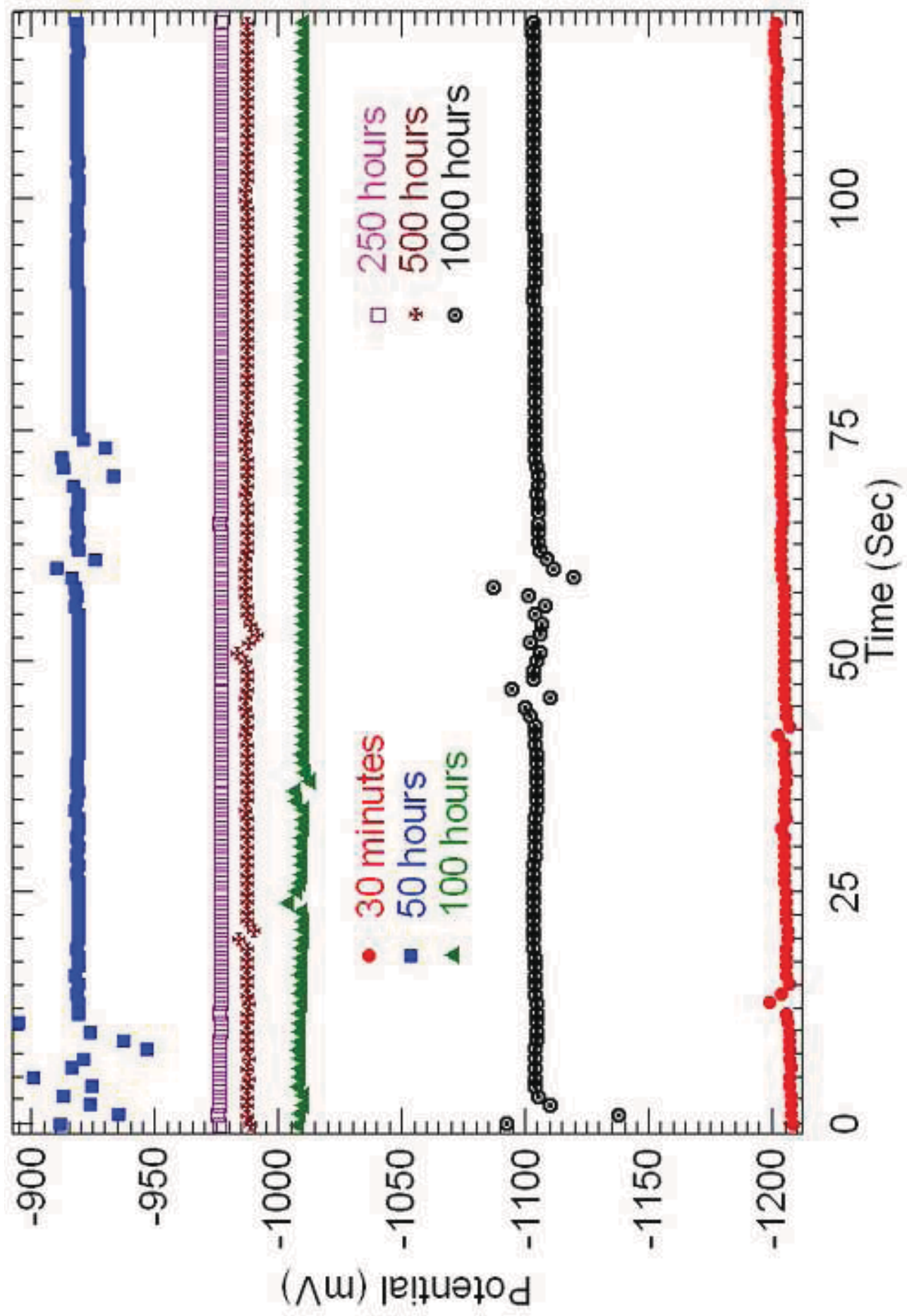


Figure 11
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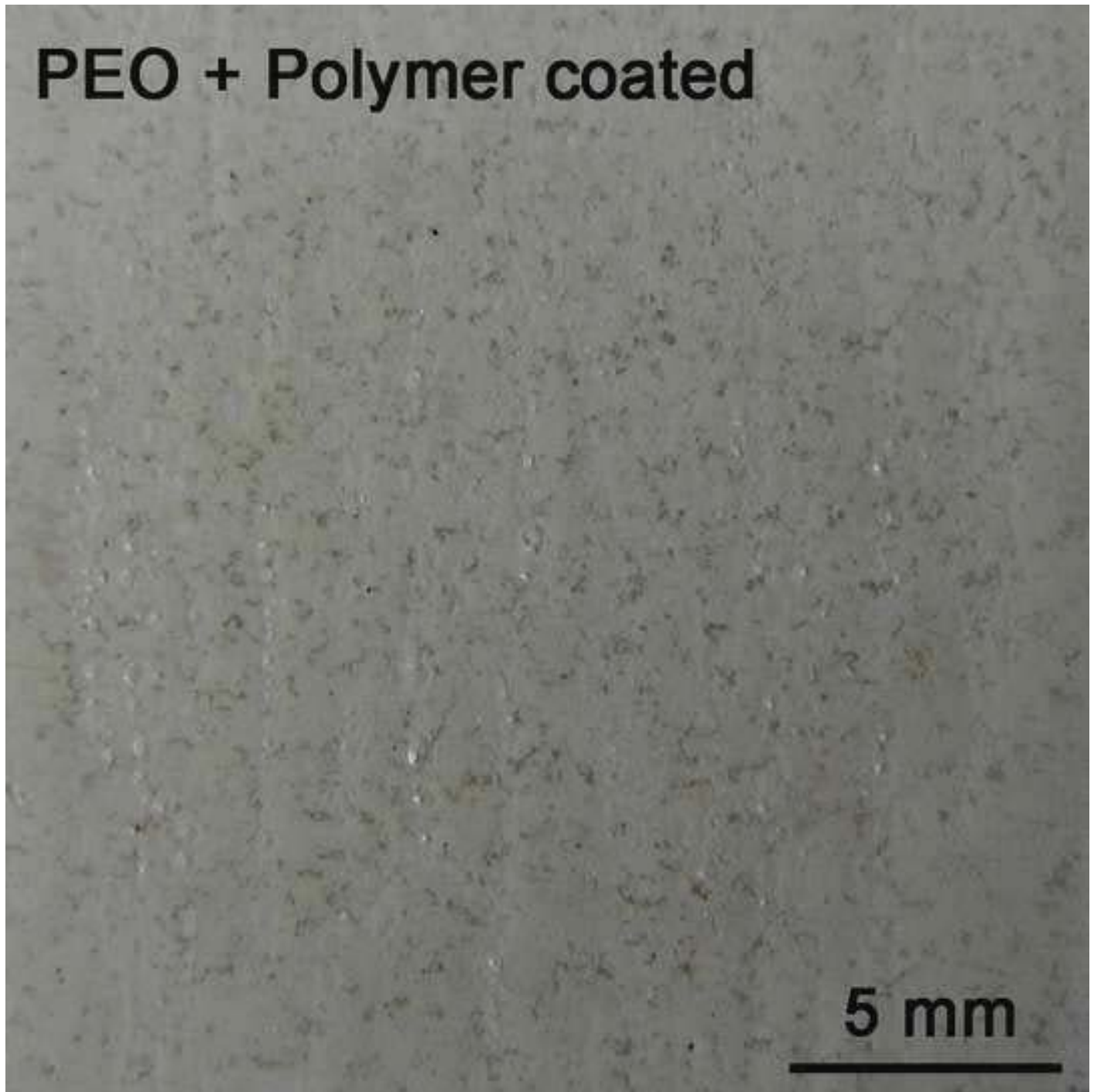


Figure 12a
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Figure 12b
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Figure 12c
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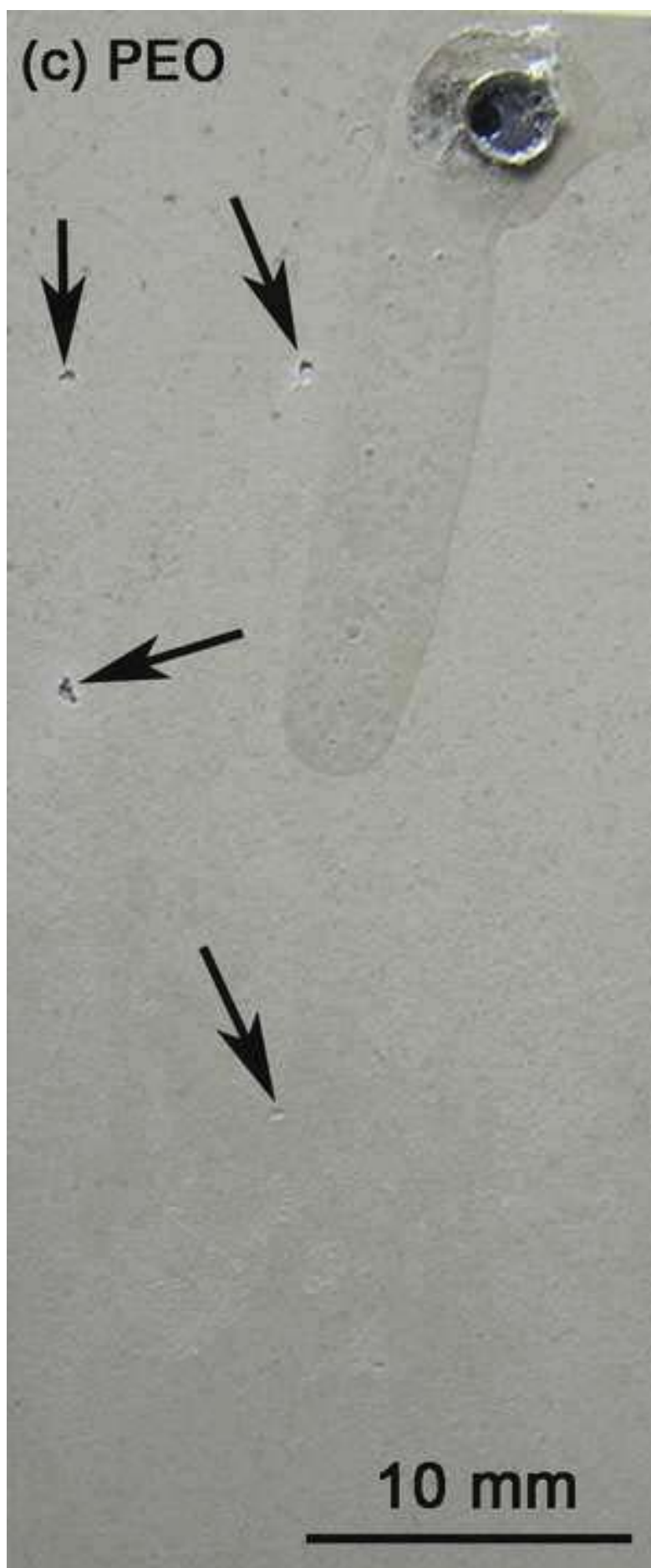


Figure 12d
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